LLNL STEMMING PLUG ADVANCES

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DNA 2nd Containment Symposium
Albuquerque, New Mexico

July 29, 1983

Lawrence Livermore National Laboratory

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ABSTRACT

In early 1982 the Lawrence Livermore National Laboratory (LLNL) conducted its first examination of in-place rigid Coal Tar Epoxy (CTE) stemming plug material. That evaluation, and subsequent downhole samplings, cast uncertainties on the emplaced strength characteristics of CTE plugs. A number of replacement plug materials were evaluated, and a two-component epoxy system was chosen for in-place testing in the fall of 1982. Showing high shear, flexural, and compressive strengths, and requiring relatively simple fielding techniques, this material, (denoted Two Part Epoxy, or TPE) was specified for its first application on the MANTECA event in December 1982. This first emplacement proved successful, and was followed by TPE use on the CHEEDAM event in February 1983. Details of the structural and thermal evaluations of the epoxies and prospective cement-based materials are presented.

INTRODUCTION

The Lawrence Livermore National Laboratory uses epoxy/aggregate composites as plugs, or discrete high strength components in the column of material placed around and above the experimental package in an underground nuclear test. These plugs serve several functions, but the most significant roles are as stemming platforms and gas flow restrictions. The stemming platform is expected to support the granular material (stemming) above the plug if the stemming below falls away. As an impediment to gas flow, the plug is meant to provide a low permeability restriction in the stemming column to inhibit gas flow through the cable bundle and up the hole wall surface. Additionally, at times, LLNL uses a formation-coupling plug to secure the emplacement pipe to the hole wall. This forces a buckling failure below, preventing damage to experimental hardware above. Normally, this plug is not considered to have a significant role in containment.

A typical LLNL stemming design has two TPE epoxy/aggregate plugs (see Figure 1). One is located at the bottom of the surface casing, with approximately ten feet in the casing and three feet below. The portion of the plug below the casing is meant to provide a seal to resist gas
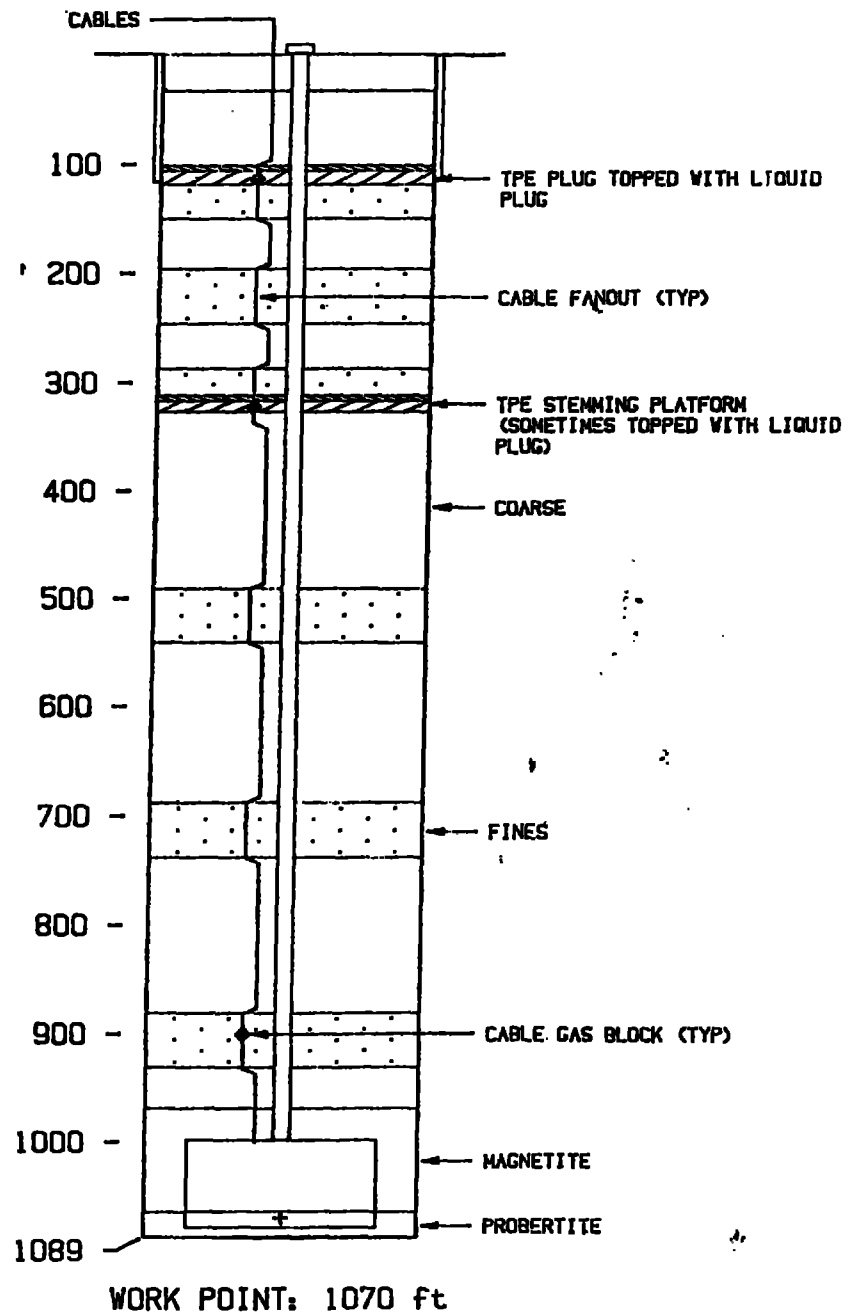
Figure 1. Typical current LLNL stemming plan.

flow outside the casing (the ultimate release path on the RIOLA event.)
The section of the plug in the casing is to remain integral in the casing
through slapdown acceleration loadings. The lower plug, or stemming
platform, is expected to survive the initial ground shock loading and
support the stemming above if either subsurface collapse or stemming fall
removes the stemming below. The stemming platform is usually keyed into
the formation with intentionally cut washouts.

Coal Tar Epoxy (CTE) has been used by LLNL for thirteen years in a
variety of formulations as stemming plugs. The fielding of these plugs
required the artful proportioning and mixing of as many as six different
components in concrete mix trucks and then dispensing the material either
directly into the hole with the mixed aggregate or pumping the liquid
through a pipe to the plug elevation and dumping the gravel into the hole
simultaneously. While the CTE plugs in general appeared to perform as
expected, their erratic thermal performance and occasional disfunction
(e.g., evidence of plug slippage in the surface casing) caused concern
that, because we rarely knew if a plug was challenged by radiation in the
hole, these features may not be performing as well as desired.

The RIOLA leakage of 1981, and subsequent investigations into the cause
of the release, indicated that the stemming platform in that emplacement
hole had in fact failed and was no longer there.

RIOLA stimulated LLNL (and LANL) to carry out experiments to evaluate the
characteristics of the CTE material as placed in a deep location. The
intense scrutiny, including tests of recovered plugs, identified real
problems with procedures, quality control, and quality assurance
practices, and highlighted some fundamental problems and pitfalls in the
basic material. In addition there were growing concerns for health
hazards with some components of CTE. LLNL at this time chose to replace
CTE with an improved material in order to eliminate the problems that had
been identified. A discussion of the tests done by LLNL on CTE is
included in Appendix A.

THE SEARCH FOR A NEW MATERIAL

A small effort had been directed toward a new material during the preceding couple of years, encouraged by the potential for lower cost and to reduce the health hazards mentioned above. One was a polyester concept that was judged to require a great deal of development work, particularly for deep-hole emplacements of large quantities. The other, which can be called the origin of the material now in use, focused on modern epoxy materials being brought into commercial use for coating applications. These seemed to have some desirable features, such as a reduced health hazard, the capability to cure and bond in moist environments, and simplified fielding procedures.

The general requirements were easily defined at that time. Already established was a minimum strength goal which was clearly feasible in the epoxy-gravel mat:ials. Field simplicity was a strong requirement and a lesser problem in health hazards was desired. The maximum temperature in curing was to be less than 150^0 F to prevent damage to downhole cables. Due to the observations of the recovered plug materials, it was decided at the start that the plastic and the gravel would be completely mixed before sending the materials into the hole. Emplacement by means of a small diameter pipe was seen as a desirable feature of the material.

Laboratory tests were done on small batches until a good candidate formulation was identified. At that point, plans were made to go to the field for emplacement of a test plug which was to be recovered and examined in detail. That material is the same formulation that LLNL now uses at NTS--TPE, for Two Part Epoxy. It is known by the Celanese Corporation as RDX-60366 and RDX-60367, an epoxy and its hardener, respectively. Preparation in the field involves mixing equal volumes of each of the two components (a procedure that works very well due to its obvious simplicity), combining with sand and gravel, mixing and dispensing into the emplacement hole.

The details of the testing program are discussed in the following sections and in Appendix B. As things have turned out only one of the early objectives was not achieved. Emplacement through a small pipe is not currently feasible. In all other respects the material is working out well for field operations, and has instilled enough confidence that LLNL is using shorter and fewer plugs than in the period following RIOLA.

TWO PART EPOXY MATERIAL EVALUATION

Strength of the material at shot time is of major importance; this can be as soon as 30 hours after the last plug is completed. Factors to consider are the degree of cure, actual temperature, laboratory measurement of strength and the degradation of strength in emplacement. TPE requires 24 to 48 hours to reach the fully-cured condition when mixed with the gravel to form a plug composite, and the temperature at the contact with the surface casing is the dominant interest. The strength of the epoxy is strongly affected by elevated temperatures.

After the Celanese material was selected by LLNL, two test plugs were poured and retrieved for strength determination in September, 1982. The first was poured directly from the surface into an 88" diameter can, which was suspended at the 1000-ft. depth in a 96" hole (U2fe). The second was emplaced in a 48" can at 600 ft. by pouring the epoxy/aggregate mixture through a 4.5" pipe to the plug elevation. The epoxy/aggregate ratio was the same as had been used in CTE emplacements in the past, filling the voids in quartz gravel (approximately 40% void fraction with the epoxy.) In volume parts, then, the mix proportions were one part gravel to .4 parts epoxy (giving a total volume equal to the volume of the gravel.) The results of the structural evaluations on the retrieved material are shown in Table 1.

The first plug as it is being separated from the can is shown in Figure 2.

TEST	NO. SAMPLES	AVG. STRENGTH (psi)	STAND. DEV. (psi)
Flexural (dumped into hole)	4	1001	92
Flexural (Poured through pipe)	4	967	184
Flexural (QA results)	3	1455	163
Compression (.0001/s strain rate)	1	1334	--
Compression (.05/s strain rate)	1	1276	--
Compression (.1/s strain rate)	1	1972	--

Table 1. Two Part Epoxy retrieved plug results.

Two issues are worth noting with the strength test results. The first is that the downhole flexural strength is approximately 70% of that expected from the laboratory and quality assurance tests (the Coal Tar Epoxy downhole plug material proved to be only 25% to 30% of its QA strength). The second point is that emplacement through the 4.5" pipe had no significant effect on the strength of the downhole material, and visual examination of the retrieved plugs gave no evidence of foreign material intrusion or layering. In general, for TPE and CTE plugs mixed completely at the surface and dumped into the hole, LLNL has no evidence that this emplacement technique grossly affects the homogeneity of the plugs.

Once the plug tests showed positive results, it was decided to proceed with the field emplacement of the new epoxy on the MANTECA event, to be stemmed in December, 1982. Plans were to place a deep plug (375' (/n) through a small pipe, about 4.5" diameter, then later place the plug in the surface casing by dropping it directly from the surface.
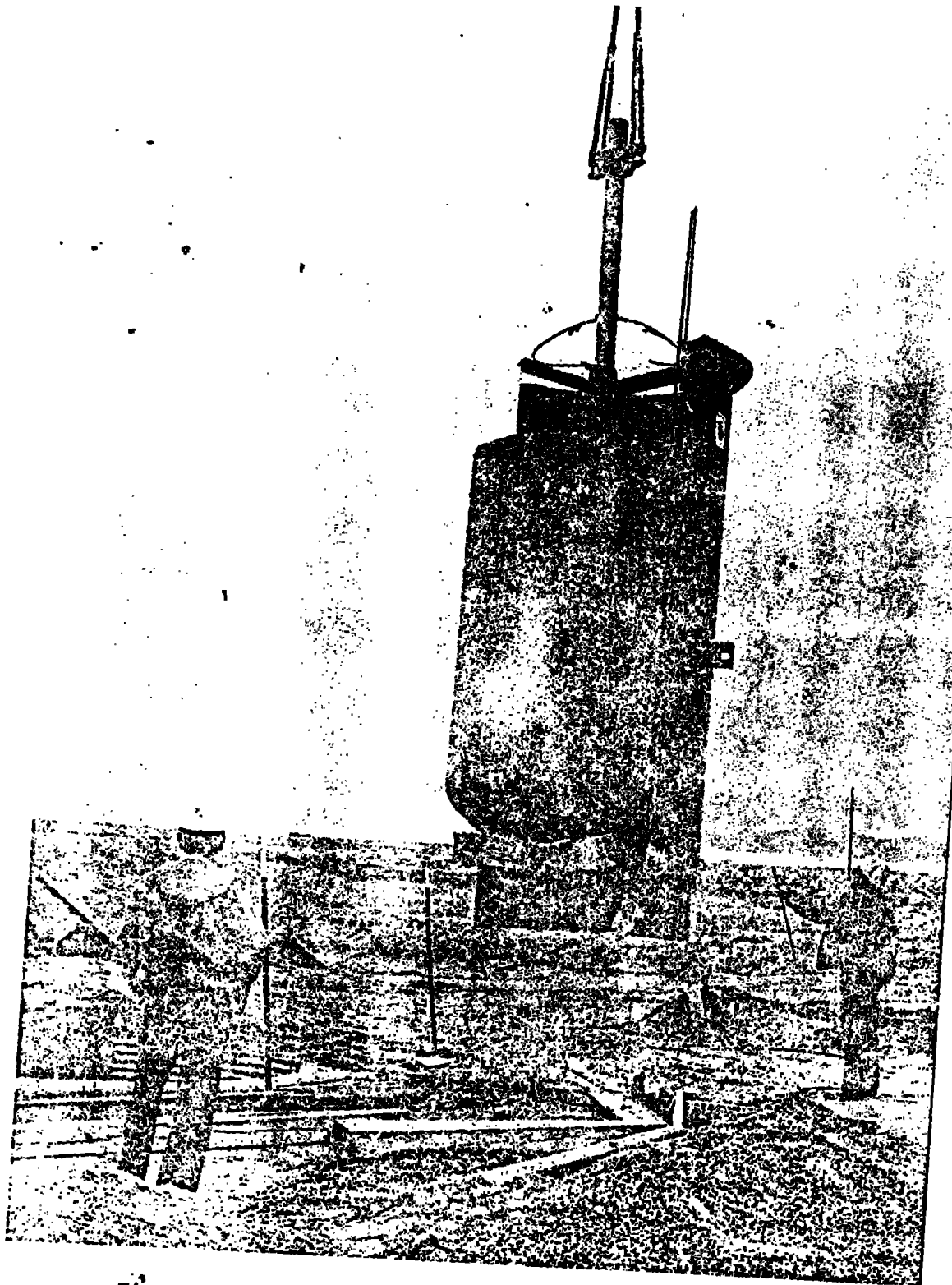
Figure 2. The first U2fe test plug being extracted.

Conductivity:	Temp. (F)	Cond. (Btu/hr-ft-F)
Unfilled TPE:	86	.125
	138	.123
	188	.126
TPE Filled w/Gravel:	86	.849
	135	.826
	185	.794

Specific Heat (TPE Only):   .592 @ 176 F

Specific Heat of Overton Sand: .200 @ 176 F

Specific Heat of Quartz Gravel: .205 @ 176 F

Epoxy heat of reaction: 113 Btu/lb

Table 2. Thermal properties of TPE and aggregate.

As discussed above, the test of putting mixed material down through the pipe was successful. In the MANTECA operation it did not work out at all. The early test was a small batch and was noticeably aerated by the mixing process. The MANTECA plans reverted to the straight dumping process as had been done on prior events with CTE, and as had been done on one of the TPE tests. No problems were observed and this has become the LLNL method of placing TPE.

The testing and thermal property measurements that followed indicated that earlier attempts at measuring the temperature rise were probably not of sufficient size or of the appropriate configuration to create an adiabatic environment, a condition which the center of the plug represents., The thermal characteristics of the TPE and aggregate material are shown in Table 2. With this information, the temperature rise seen on the MANTECA plugs was in fact consistent with the properties of the components.
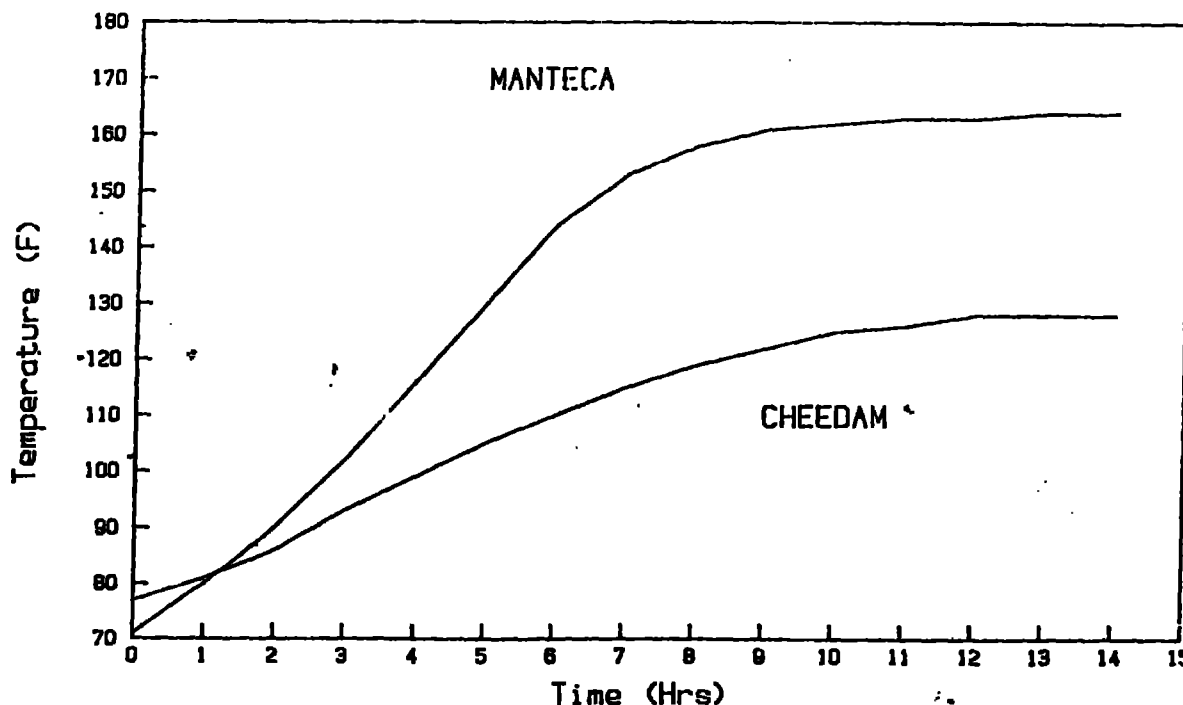
Figure 3. Temperature responses showing the influence of the aggregate changes after MANTECA.

To reduce the temperature rise to a more acceptable level, a number of modified aggregate formulations were tested. The regular quartz gravel was supplemented with silica sand (Overton sand) in different ratios, and sand alone was tested as an aggregate. The epoxy/sand formulation was ruled out because, while it poured easily through the emplacement pipe, it exhibited unacceptable shrinkage during the cure process. The sand/gravel aggregate formulation that was chosen for further testing was a combination of 3 parts quartz gravel to 1 part sand, combined with .75 parts epoxy (to make a total volume equal to the volume of the gravel). With this formulation, the expected temperature rise was 51 F^O, compared to the 90 F^O expected (and measured) on the MANTECA formulation. No changes in the epoxy itself were made.

This fix was verified with a test plug pour in U9y24 in February, 1983, and subsequently fielded on CHEEDAM and later events. Figure 3 illustrates the temperature history of the two epoxy/aggregate formulations, comparing the MANTECA and CHEEDAM temperature responses. Emplacements since CHEEDAM have followed that general 50 F^O temperature rise.

Full Cure Strength of 3:1:.75 Mix of Gravel, Sand, TPE:
(5 day cure @ ambient, 20 hrs. @ temp.)

	Temp. (F)	Strength (psi)
Compressive (Meas.)	80	1260
	100	580
	120	350
	140	210
	160	160
Direct shear:	80	630
(.5 x compr. str.)	100	490
	120	175
	140	105
	160	80

Degree of Cure: Insulated compressive test samples indicated
         approximately 60% of full cure strength at 24 hrs.
         and 100% at 48 hours (referenced to full cure at temp.)

Table 3. Mechanical properties of the TPE/aggregate mix as functions of
          temperature.

Pertinent structural properties for the modified epoxy/aggregate
formulation are provided in Table 3. By adding sand to the quartz
gravel, it has been possible to reduce substantially the epoxy required
to bond the aggregate together, reducing the temperature rise by 33%,
reducing costs of emplacement, and maintaining the high structural
properties. This modification, however, has made the mixture
sufficiently less liquid to rule out any possibility of emplacing it
through a small diameter pipe to the plug elevation. This does not seem
necessary in light of the negligible increase in strength seen in the
U2fe test plug emplacement through a pipe, compared with the previous
emplacement by dumping directly into the hole.

The expected variations in the void fractions and particle size distributions in the gravel and the sand has led us to a revised formulation that is nominally a little rich in liquid, with epoxy about 10% greater than the mix used in CHEEDAM. Volume proportions of gravel:sand:epoxy are now at 3:1:0.83 in current applications.

ADVANCED CONCEPTS IN STEMMING DESIGN

The new epoxy plug material development program has enlightened the LLNL Containment community with the realization that polymer plug materials are sensitive to environmental conditions, costly and difficult to emplace, hazardous to workers, and susceptible to strength reduction from their own heat of reaction. In the process of evaluating the new material and alternative emplacement concepts it was clear that there was virtually no experience outside the Nevada Test Site pertinent to the emplacement of epoxy/aggregate concrete in such massive concentrated volumes. While the Celanese epoxy was essentially derived from products used in the coatings industry, there was little information that could be extended to nuclear event applications.

In a parallel, but much smaller effort, LLNL was evaluating cements as possible plug materials. Combining the understanding of the polymer problems and some knowledge of cement materials, it was decided to wipe the slate clean and consider the ideal way to fill an emplacement hole for containment of a nuclear event.

In its simplest form, the emplacement hole would be completely filled with a material that would act like the local medium and have enough strength to remain in place through the various collapse scenarios. It would also be of low permeability, would not damage cables, be quick and inexpensive to emplace, and be a reliably performing material. While filling the emplacement hole entirely with one good material is a simple design, it would be too costly.

The two regions of concern in stemming design are the stemming directly above the experiment and the upper region of the emplacement hole. The region directly above the canister is of interest because it is in that range during low yield events that a competent material will enhance the integrity of the residual stress field and maintain the containment cage. This has the effect of preventing early time gas flow up the .stemming column. The upper region of the hole is a critical area in that it is the last defense should a subsurface collapse .eliminate the lower portion of the stemming column. Consequently, it is desireable to have a containment feature in the upper hole that will remain effective throughout any of the collapse scenarios, acting as a gas blockage as well as a structural member.

LLNL is pursuing actively a stemming design concept that addresses directly both the prevention of early time gas flow and the integrity of the upper stemming column. The initial concept is shown in Figure 4. It consists of a feature extending from the top of the diagnostic canister out to a range of 1.75 to 2 cavity radii for yields up to perhaps 12 kt. It is sized large enough to accomodate uncertainties in yield and in the location of the region of high residual stress (estimated at 1.25 cavity radii), and is to have properties that allow it to enhance the stress field rather than provide the discontinuity inherent in low strength, highly compressible granular stemming material. The upper features are long (perhaps 40 to 50 ft.) fairly strong components designed to remain in place through ground shock and slapdown accelerations and provide multiple redundancy to subsurface collapse.

Most promising at this point is a concrete consisting of gypsum cement and coarse aggregate (tuff fragments from the NTS shaker plan). This combination has several advantages:

1)   Gypsum cement achieves full strength in less than one hour.
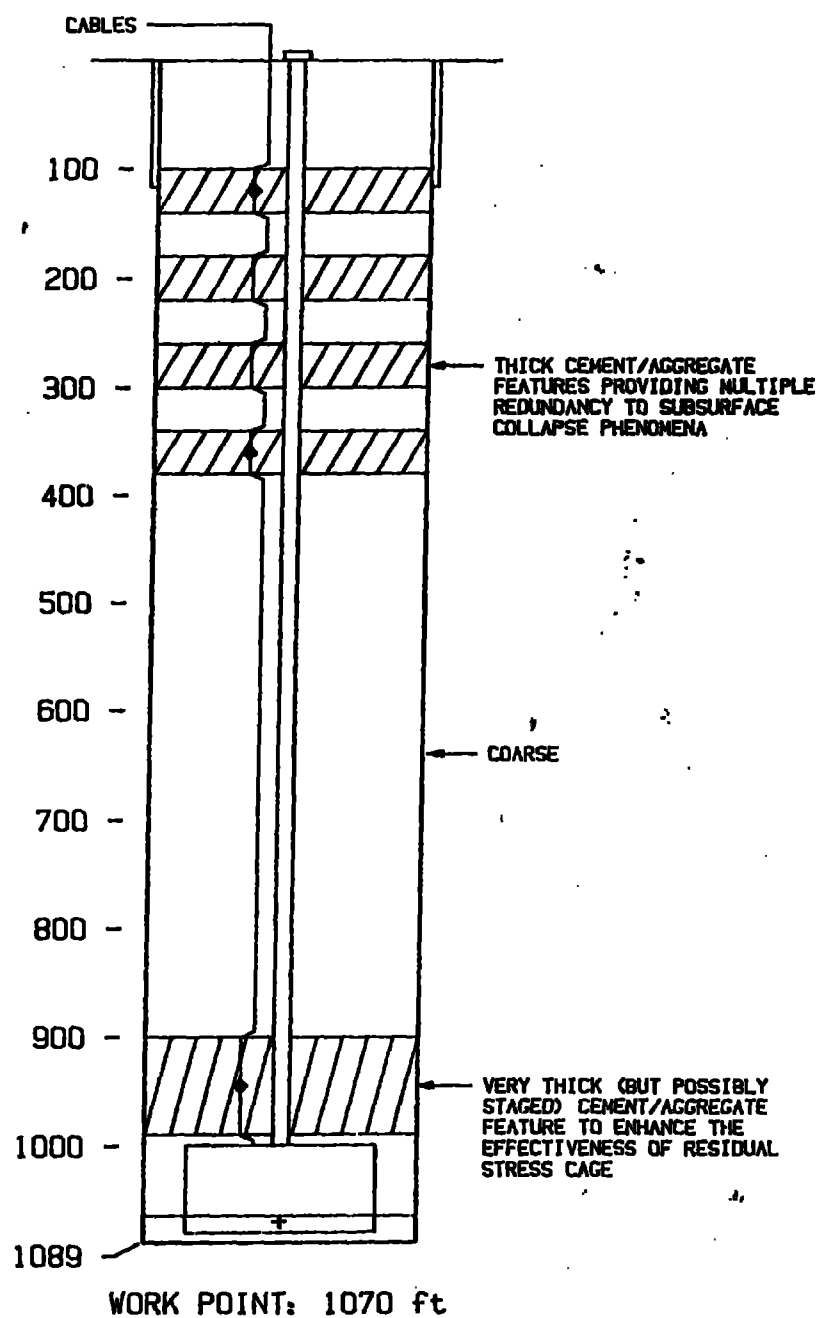2)   The gypsum slurry is of very low viscosity, and flows easily around cables and downhole hardware.

CABLES

100 —

200 —

300 —

THICK CEMENT/AGGREGATE
FEATURES PROVIDING MULTIPLE
REDUNDANCY TO SUBSURFACE
COLLAPSE PHENOMENA

400 —

500 —

600 —

COARSE

700 —

800 —

900 —

VERY THICK (BUT POSSIBLY
STAGED) CEMENT/AGGREGATE
FEATURE TO ENHANCE THE
EFFECTIVENESS OF RESIDUAL
STRESS CAGE

1000 —

1089 —

WORK POINT: 1070 ft

Figure 4. The proposed stemming design using large cement features.

3) Gypsum expands linearly .3%, enhancing coupling to the formation.

4) The strength of the cement/aggregate concrete is reasonably high, in the range of 1000 to 1300 psi in laboratory tests.

5) The material has numerous fielding advantages, including low heat of hydration, relatively low cost, ease of emplacement, no significant health hazards, no strength sensitivity to exotherm temperatures and most importantly, a broad industrial experience base from which to draw.

Recent fielding tests by LLNL have evaluated emplacement methods and equipment. The most probable technique will involve simultaneously pumping gypsum slurry through tubing to the plug location while dumping the aggregate material into the hole with conventional stemming equipment.

The rates of emplacement possible with this approach suggest that total stemming times could be reduced by as much as 60%. While the cost savings can be significant with this approach, the confidence gained by emplacing as much as 250 ft. of "plug" material (compared to the current designs utilizing 23 to 25 ft. of epoxy plug material) is substantial. The projected plans for the emplacement of this design include laboratory and analytical evaluations of the concept's effectiveness, emplacement of the upper features as an addition in a satellite hole, emplacement of the lower plug feature on events with appropriate diagnostics, and eventually fielding of the complete stemming design in the second quarter FY 84.

SUMMARY

The CTE evaluation program illuminated several problems with materials and techniques in plug emplacements. Development of the Two Part Epoxy system, with the modified aggregate, has provided a plug material that has shown good downhole strength, consistent thermal behavior (providing some indication of consistent emplacement), repeatable structural characteristics, and ease of fielding. The new material has instilled sufficient confidence that LLNL has been able to use fewer and shorter plugs than in the past.

However, with this greater understanding of epoxy characteristics, it is clear that there are many problems associated with the emplacement of polymer plugs in stemming columns, due primarily to their sensitivity to environmental conditions and mixing techniques. Coupled with these concerns is the uncertainty of the usefulness of a small number (2 or 3) of short discrete structural plugs in the stemming column when the predictability of subsurface collapse is imprecise. The new concepts in stemming design address these uncertainties with several large cement/aggregate features to provide multiple redundancy, and a feature in the lower region of the hole designed specifically to enhance the capability of the low yield events to essentially contain themselves in early times.

These concepts have the potential of providing increased confidence in the engineered containment fetures, while simplifying and shortening stemming operations.

ACKNOWLEDGEMENT

The authors and the Nuclear Test Engineering Division would like to acknowledge the extensive development work performed by Phil Fleming in the early stages of the new material evaluation program.

APPENDIX A: COAL TAR EPOXY EVALUATION

Three separate tests were carried out with the intent of evaluating the coal tar epoxy plug materials in place at depth. The investigations were planned to provide insights into the basic material, the emplacement methods, or any other factor of the downhole environment.

The first test was done in hole U4ac with fully mixed material (epoxy and gravel) poured to a depth of 788 feet into an instrumented container forming a plug 88" in diameter and 10 feet thick. The second and third tests were done in conjunction with the demonstration of LLNL's Cable Downhole System. Because there was no nuclear device but it did have an array of cables, these tests had the potential of providing more information than just on material properties. Large samples were retrieved with long, conical slotted buckets, pulled out of the plugs before significant strength developed, but late enough that the material remained in the buckets. In addition to visual observations of the material, a number of tests were done to evaluate mechanical properties, degree of cure, and possible contaminants. Samples were taken for QA purposes in the same manner as for the nuclear events to compare with the downhole samples. Numerical results are given in Table A1.

Observations from U4ac

Visual inspection showed that the material was well mixed with no indications of layering or gross contamination. Flexural tests and shear tests produced results well below the values expected. Infra-red spectral analysis indicated that the epoxy had not reached a state of full cure. Review of all procedures and tests of the QA samples showed no deviations from normal event practices and experience.

Sample	Flexural Strength (psi)	Direct Shear (psi)
U4ac Downhole	284	103
U4ac QA	857	n/a
LLNL History	630	n/a
CDS Retrieved Mat'l.	262	n/a
Laboratory Samples	n/a	577

Table Al.   Coal Tar Epoxy retrieved material properties.

Observations from the CDS Tests

One of these tests was done by fully mixing the material at the surface and dumping directly into the hole, while the other had liquid pumped to the plug location through tubing with the gravel added from the stemming conveyor. · The fully mixed material remained mixed at depth, while the pumped plug exhibited severe layering of aggregate and epoxy. Structural tests again showed strength to be significantly degraded from expectations.

Other Observations

The tests done on CTE afforded opportunities to evaluate the procedures, equipment and QA methods outside of the schedule pressure of a nuclear event. In addition, the ongoing event series did allow some testing of samples made up in addition to the normal set required. Several things

became quite clear:

1) The CTE formulation appeared sensitive to environmental and fielding conditions: moisture, initial temperature, mixing orders of components, etc.

2) Mixing of the CTE components required overly precise control for the field conditions.

3) The QA procedures were not capable of identifying problems with mixing procedures and tended to mask small problems with the formulations.

These problems made it clear that it was time to find a new material.

APPENDIX B: TPE AND GYPSUM CEMENT TESTING TECHNIQUES

It was decided early in the material testing program to consider the epoxy/gravel mixture a concrete and test it as such. Some effort was directed toward identifying a single standard test (preferably ASTM) that could reliably characterize the most important strength properties of the plug material (compressive, tensile, and shear strengths). The same tests were applied to the cement materials. Pictures of a number of these test specimens, both epoxy and cement, are in Figures B1 through B8.

A series of tests was conducted on the TPE/gravel formulation used on MANTECA (1 part gravel, .4 part TPE), to obtain enough data to determine if the ratios between the various tests were constant within normal data scatter. Data are presented in Table B1 for samples at ambient temperature, tested at least 10 days after they were poured.

The tests used were as follows:

1.   Compressive test ASTM C-39

TEST	NO. SAMPLES	AVG. STRENGTH (psi)	STAND. DEV. (psi)
Compression	5	2584	21
Direct Tensile,	5	603	59
Split Tensile	5	565	8
Flexural	5	1353	89
Double Shear	5	1522	190
Punch Shear	3	1303	100
Pull-Out Shear	20	1200	60

Table B1.   TPE mechanical properties comparing test methods.

2.   Split tensile ASTM C496

3.   Direct tensile - ASTM D2936

4.   Flexure ASTM C78

5.   Double shear 2" x 2" x 10" Western Technology Inc. custom tooling

6.   Pull-out shear ASTM C900-38T

7.   Punch shear 3" hole with 2.5" D punch, 3" thick sample.

A total of 130 mechanical tests were made.   The data presented is a representative sample of these tests.   Based on this testing program, the following conclusions can be made about TPE and pea gravel concrete:

1.   The compressive strength of the material is probably the best all around measurement if only one type of test is to be made.

2.   The tensile strength appears to be about .2 times the compressive strength.

3.   The shear and flexural strength is about .5 times the compressive strength.

Similar tests were conducted on the gypsum cement, along with measurements of peak temperatures. It appeared that gypsum cement with or without filler rock could meet the LLNL temperature requirements.
U S Gypsum W-60 or Halliburton "Cal Seal", two commercially available gypsum cements, were reported to have a heat of hydration of 47.8 Btu/lb. and a specific neat of .22 Btu/lb F^O . With a mix ratio of 100 lb W-60 to 40 lbs of water the calculated temperature rise would be 76 F^O. This would be the highest value that could be reached assuming a good mix of gypsum cement and water and no separation during emplacment. Any addition of rock, gravel or gypsum gravel would result in a lower temperature. Tests in 55-gallon insulated 'drums confirmed the temperature prediction. A number of measurements were made on gypsum cement with and without various fillers. Some general comments follow:

1)   Tensile strength and energy absorbtion are greatly increased with the addition of about .5 lb. of fiberglass per 100 lb. gypsum cement.

2)   The fiberglass strands (.5 inches long) are very difficult to blend because of wetting problems.

3)   Unfilled gypsum cement typically reaches 2500 psi compressive strength in one hour, and increases in strength as the material dries.

4)   Filling the gypsum cement with coarse fill reduces by about a 50% the compressive strength of the concrete, depending on the aggregate used.
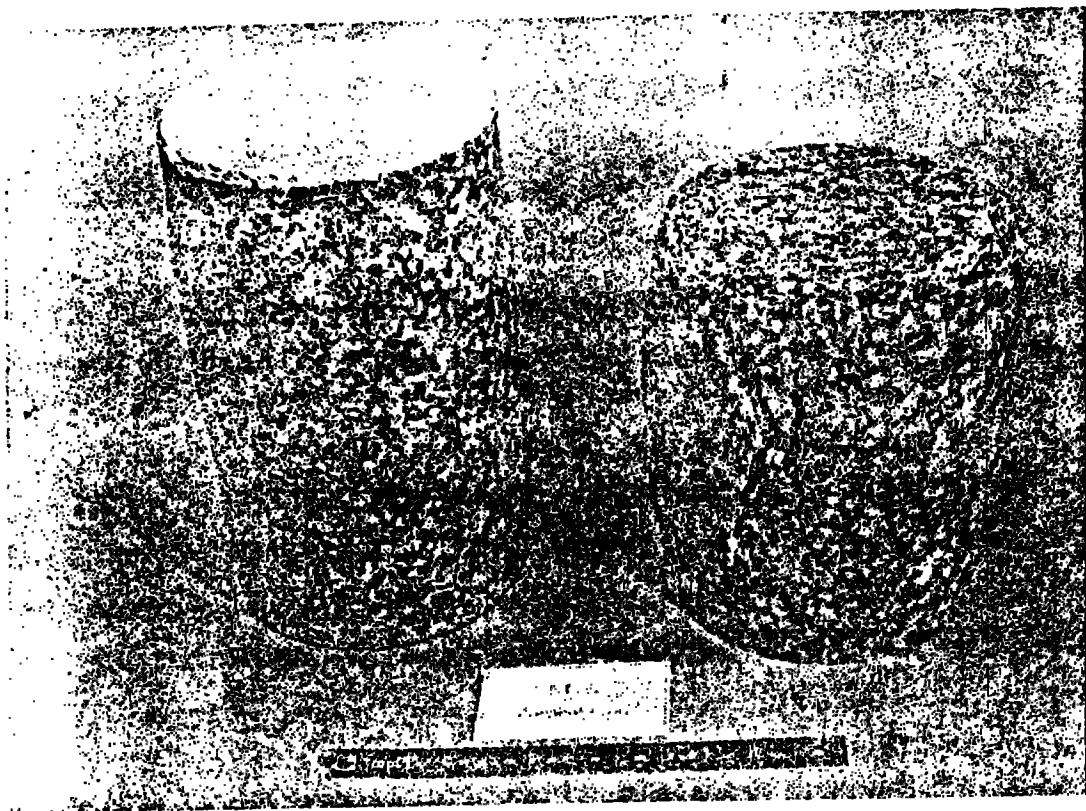
Figure B1. TPE Compressive Samples
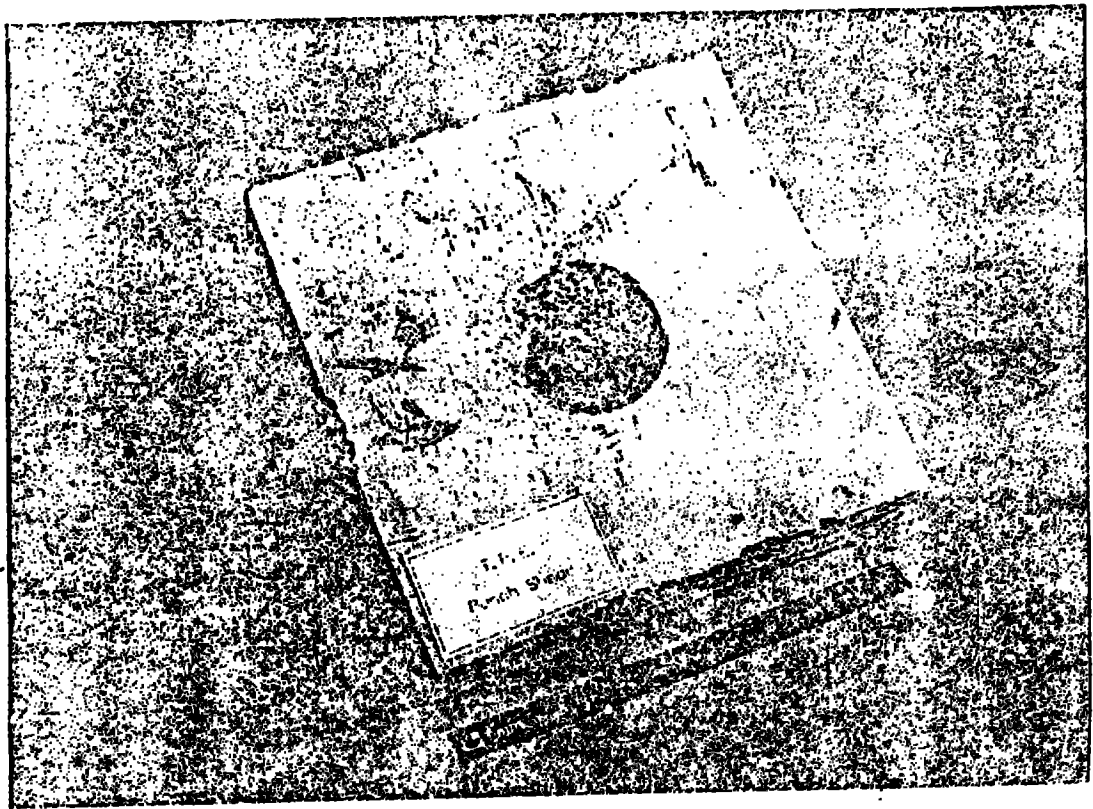

Figure B2. TPE Pullout Test Samples

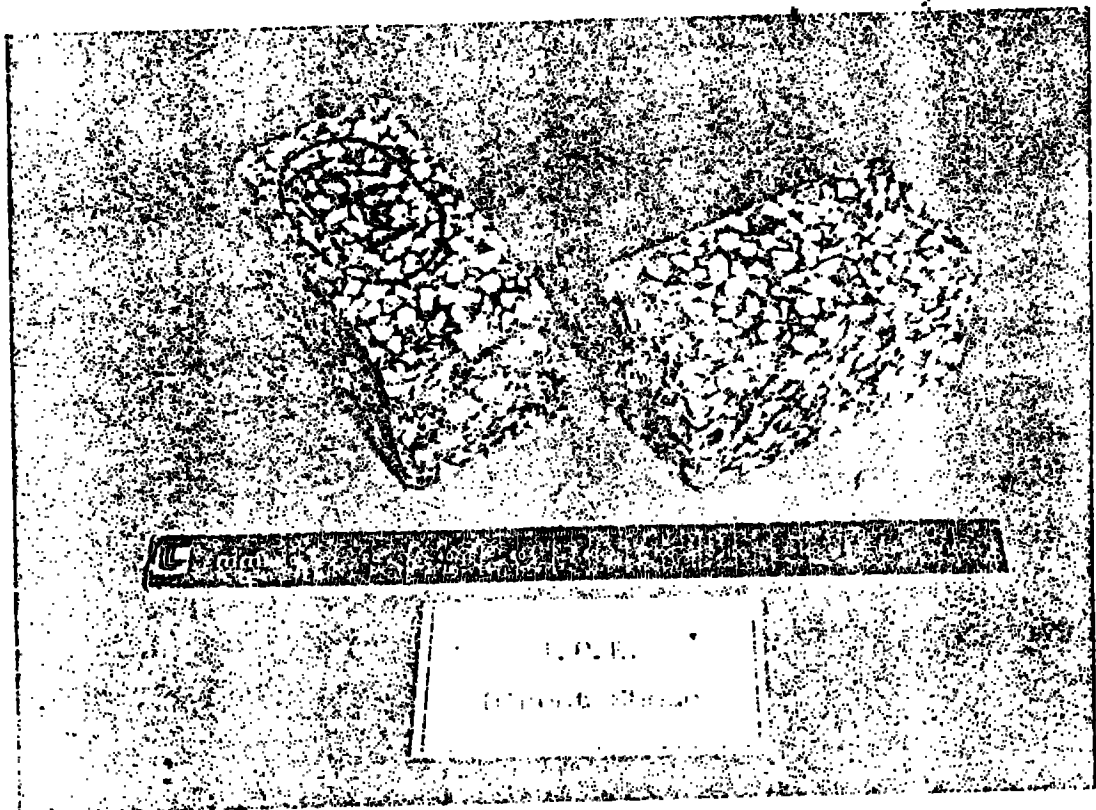Figure B3. TPE Punch Shear Test Samples

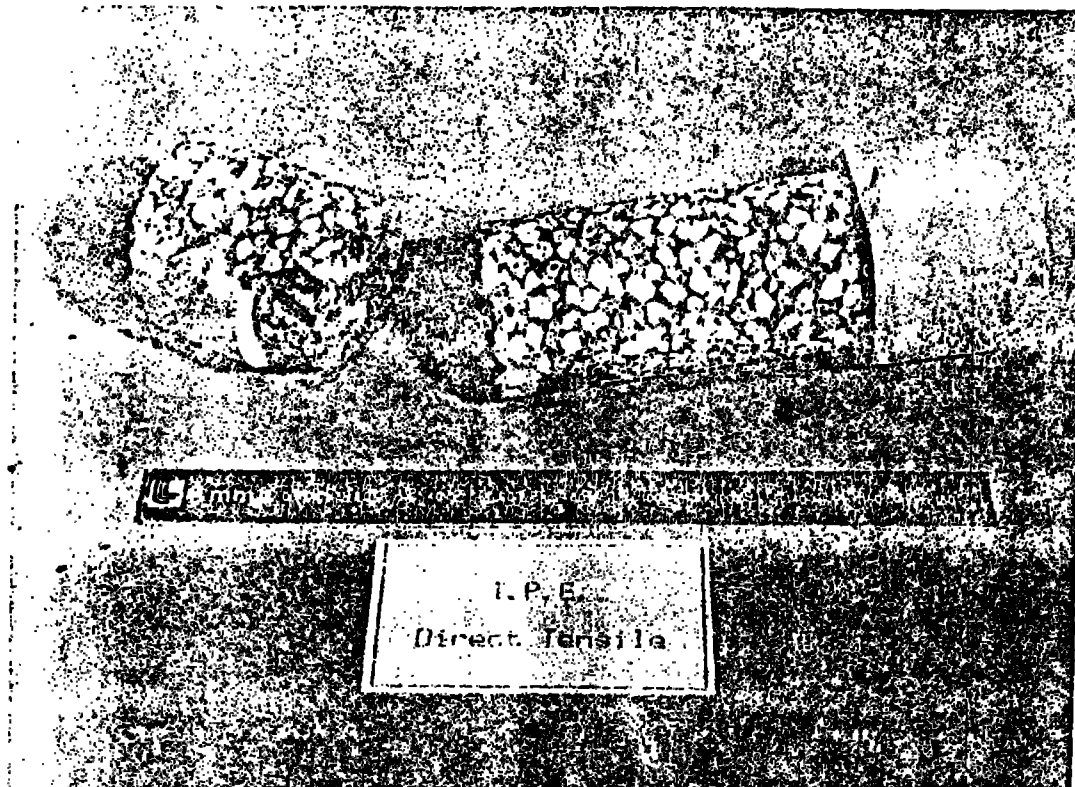Figure B4. TPE Direct Shear Test Sample
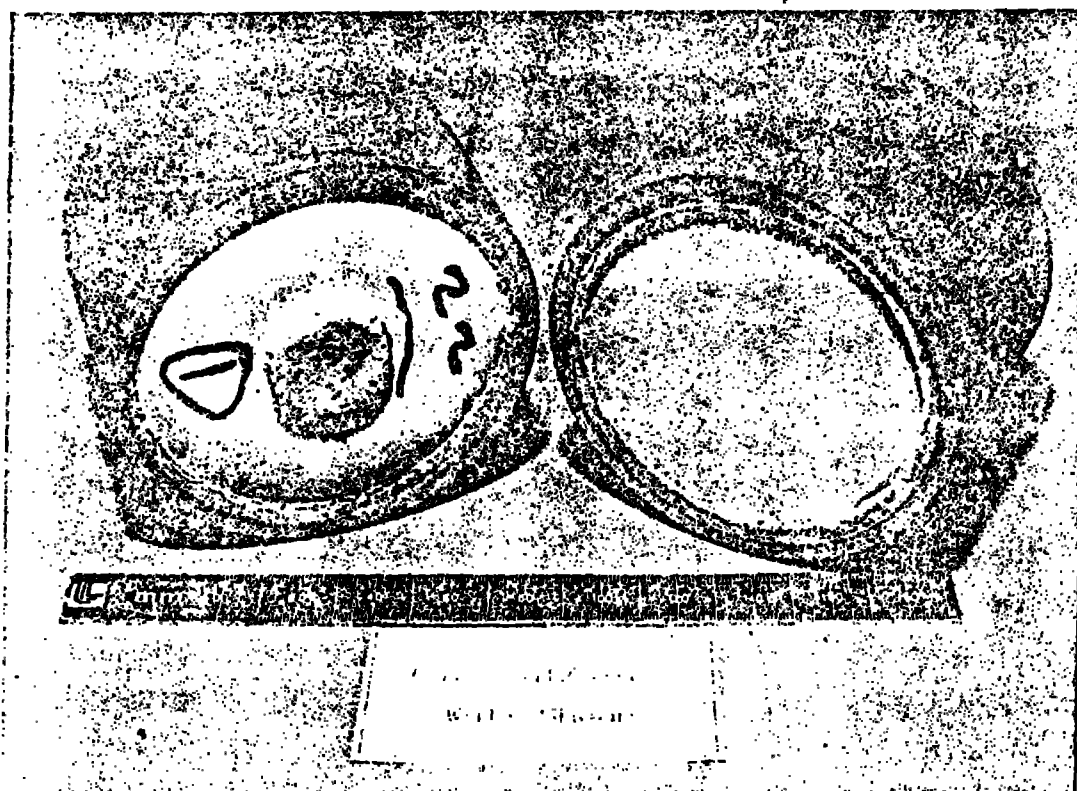
Figure B5. TPE Direct Tensile Test Sample

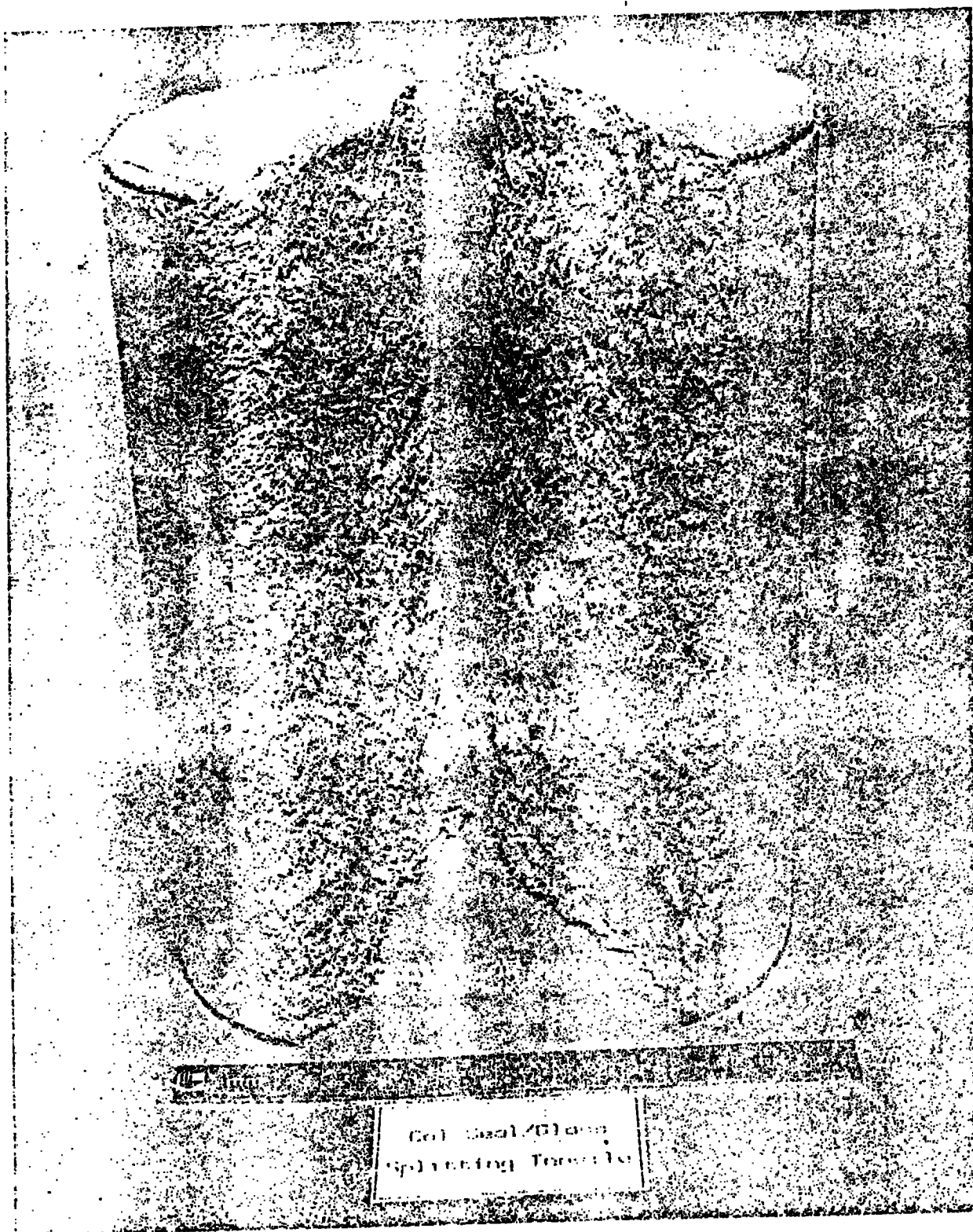Figure B6. Gypsum Cement Wall Shear Test Sample
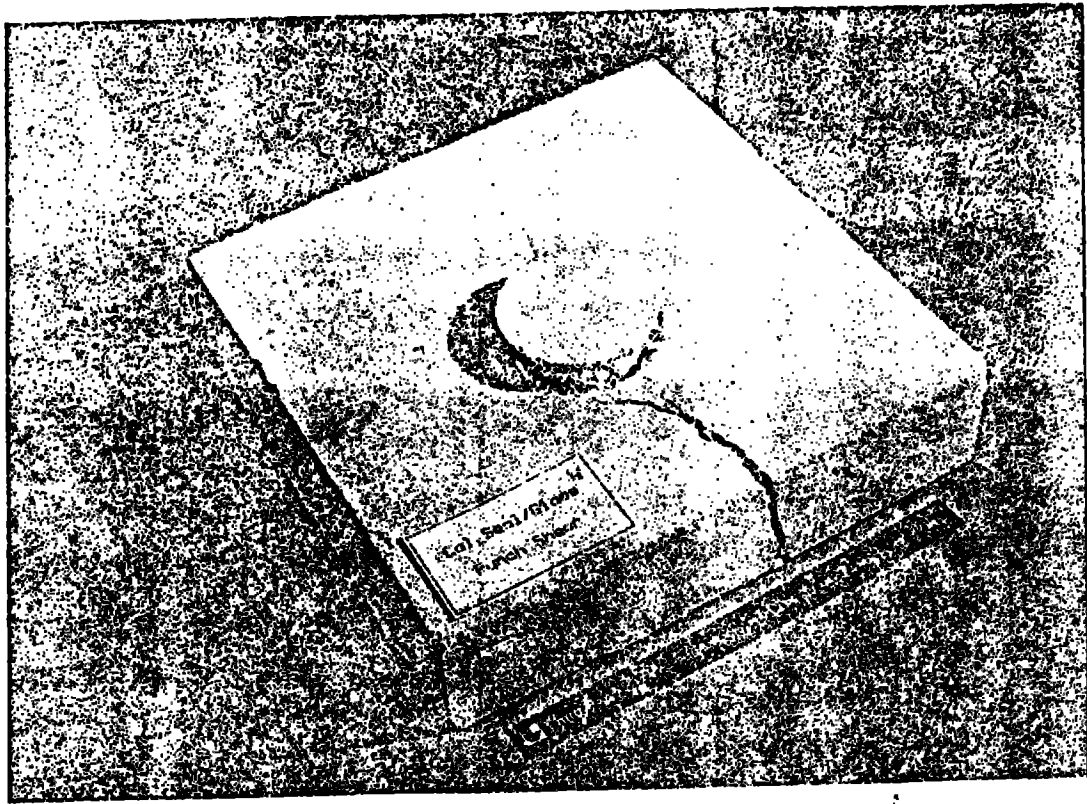
Figure B7: Gypsum Cement with Fiberglass Split Tensile Test

Figure B8. Gypsum Cement Punch Shear Test.